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Dr. Salima BOUVIER

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99, Av. J.B. Clément
Villetaneuse 93430
France

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14. ABSTRACT

This report results from a contract tasking University Paris 13 (France) as follows: The goals of this research are threefold. It is proposed to experimentally study the anisotropic work-hardening of high purity titanium at large strains (about 70% equivalent true strain). Unlike the commonly used uniaxial tensile test, the simple shear test offers the possibility to analyze the in-plane plastic anisotropy at very large strains involving severe crystallographic texture evolution. This important source of anisotropy can be investigated in connection with twinning evolution mechanism. The ability to perform finite strain cyclic loading using a simple shear test is also of great importance, in order to understand the evolution of the twinning during reverse loading. The experimental facilities at University Paris 13 (France) as the in-situ simple shear machine, allow the investigation of the evolution of the material work-hardening during loading in SEM, and provide an important tool in analyzing the interaction between twinning and slip.

15. SUBJECT TERMS
EOARD, work hardening, twinning evolution, Titanium

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Characterization of strain-induced anisotropy in Titanium at large strains under monotonic and Bauschinger loading

Master Student: N. Ben Mhenni

Promoters[1]: S. Bouvier & F. Gregori

[1]LPMTM-CNRS, Université Paris 13, 99 Avenue J.B Clément, 93430 Villetaneuse, France

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Summary

The goal of this project is to improve the scientific understanding of the behaviour of titanium based materials through a comprehensive experimental investigation of their plastic deformation. A thoroughly detailed multi-scale characterization (*i.e*. SEM observations, EBSD analysis and macroscopic texture measurements) in parallel with various mechanical tests was carried out in order to identify the physical mechanisms involved at the microscopic scale and their effects on the macroscopic behaviour such as. occurrence of Bauschinger effect, anisotropy of the yield stress and work-hardening. It was observed that twinning is activated. The sensitivity of the volume fraction of twins to the type of loading was analyzed in relation to the resulting macroscopic hardening.

In the literature, characterization of the behaviour of titanium based materials has been reported *only for simple monotonic loading conditions such as uniaxial tension or uniaxial compression*. In this study, the macroscopic mechanical response was studied for various monotonic simple shear tests as well as cyclic loading. It is worth noting that the simple shear test is an appropriate test for the investigation of the Bauschinger effect for plate materials since the problem of buckling is reduced. A comprehensive analysis of the physical mechanisms was carried out at large strains using cyclic simple shear test. Indeed, typically the amount of homogeneous shear strain that can be achieved for sheet materials (0.7 - 2 mm thickness, Rm 340MPa) can be of 100% (≈ 58% von Mises equivalent strain). Convenient and comprehensive experimental database was supplied in order to enable in the future the development of an appropriate elasto-plastic model which describes the mechanical behaviour for complex loadings of titanium based sheet materials for accurate simulations of forming processes.

Specifically, for the first time, in this study the interaction between twinning and slip mechanisms (dislocations pile-ups, internal stresses) and their effects on the macroscopic behavior and specifically the Bauschinger effect were investigated.

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Introduction

Titanium based materials deform by twinning as well as by slip. Twinning differs from slip in that it accommodates shear in only a 'forward' direction, and in that it reorients whole domains of the grain. These domains have a lamellar morphology and pose barriers to the propagation of dislocations across the grain. As a consequence, twinning leads to a change in the overall crystallographic texture of the material and therefore to changes of its macroscopic anisotropy. Such anisotropy is also manifest in the stress-strain response of the aggregate, where high asymmetry in tension and compression is observed.

Characterization of the behaviour of titanium based materials has been reported *only for simple monotonic loading conditions such as uniaxial tension or uniaxial compression*. It is well known that the twinning mechanism is highly sensitive to the strain path imposed to the material. The widely used uniaxial tensile testing characterization is not sufficient to completely characterize the material behaviour, especially in the case of materials in sheet form. Reduced ductility due to necking, occurrence of buckling during cyclic loading, are among the problems that restrict the identification of the behaviour from uniaxial tests.
Furthermore, these difficulties in performing mechanical characterization at finite strains under monotonic, cyclic, and non proportional loadings which result in a lack of experimental data for these loadings conditions prevent validation of recently developed theoretical models. In view of the application of titanium based materials in forming operations where complex loadings occur, experimental characterization of their plastic behaviour under non-proportional loadings is highly required.

The aim of the research conducted within this project is to contribute to a comprehensive investigation of the anisotropic plastic deformation of thin sheets of high purity α–Ti under monotonic and cyclic loading at large strains (about 70% equivalent true strain), where severe crystallographic texture evolution are involved. This important source of anisotropy is investigated in connection with the macroscopic behaviour of the material (stress-strain curve and work-hardening evolution) and its microscopic behaviour, in particular twinning initiation and its evolution.

Methodology and Experimental Procedures

The as-received material was in a form of crossed rolled disks of 254mm diameter and 16mm in thickness, which was purchased from Alfa Aesar Company (USA). The as-received disks have different initial textures: the one that will be designated as Plate #1 displays an orthotropic symmetry while the second disk that will be designated as Plate #2 has initially a *quasi* isotropic in-plane texture.

For Plate #1, Electron Back Scattered Diffraction (EBSD) and X-ray diffraction investigations were carried out. The plate was cut along the thickness direction into seven fine sheets of 1mm thickness as indicated in Figure 2. The simple shear samples were taken from the center part of plate and cut along 3 different directions, namely 0°, 45° and 90° with respect to the rolling direction (RD).

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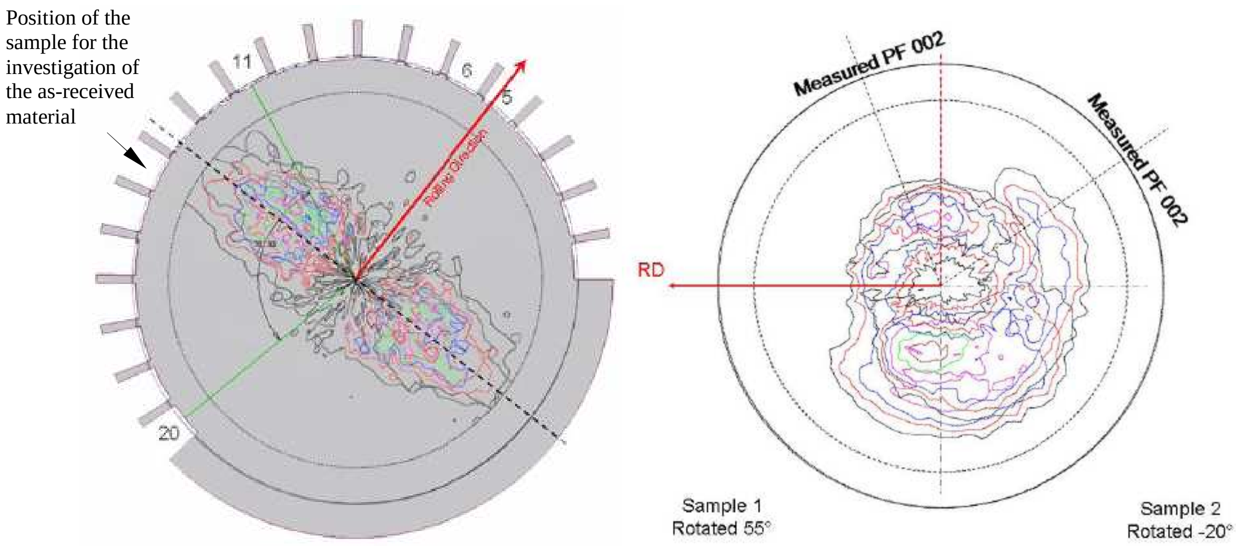

Figure 1 Basal pole (0002) of orthotropic plate#1(left) and isotropic plate#2(right).

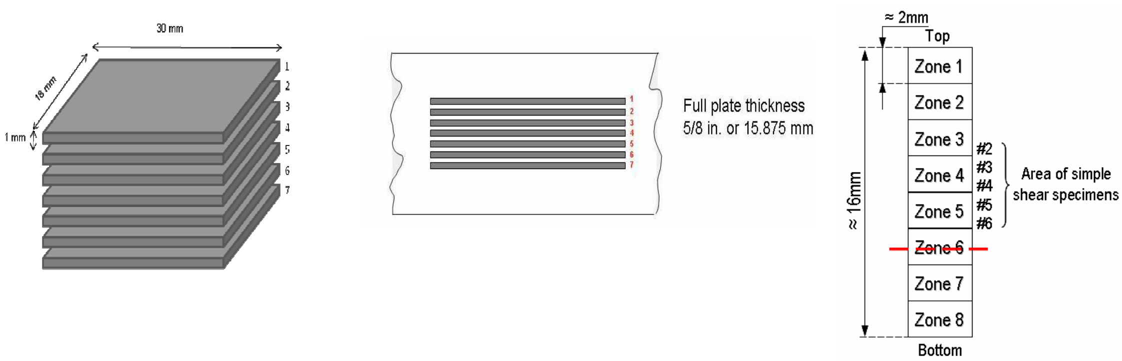

Figure 2 Specimens for simple shear.

The mechanical response was studied using quasi-static simple shear tests conducted at room temperature at a constant strain rate of $10^{-3}s^{-1}$. As compared to the commonly used uniaxial tensile test, the simple shear test has many advantages such as the simplicity of the sample geometry, the absence of any plastic instability such as localization and/or necking in tension or barreling in compression, and especially the fact that large homogeneous strains and rotations can be achieved. In addition, the loading direction can easily be reversed during the experiment by simply changing the displacement direction of the clamps, with neither dismounting the clamps nor re-machining the sample. Another advantage arises from the recording of the forward and reversed flow parameters with the same device and under the same conditions, thus avoiding some testing artifacts. Finally, the simple shear test permits to explore zones of the forming limit diagrams that are not attainable by simple tension or biaxial stretching.

In our laboratory (LPMTM), the simple shear test is carried out on a specific device put on a uniaxial tensile testing machine. Given the strain heterogeneities near the free edges of the specimens and the risk of slip under the clamping grips during testing, a great deal of attention was given to the optimisation of the sample geometry [Bouvier *et al.*, 2006a].

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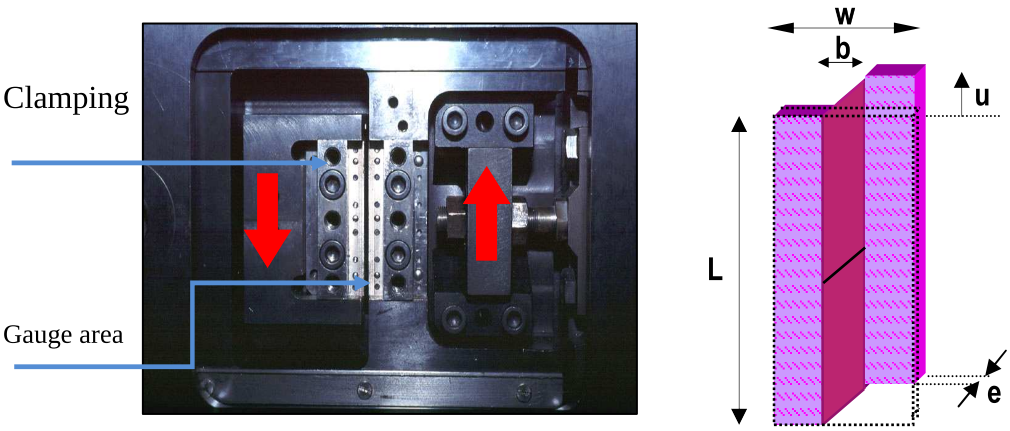
The various tests performed as part of the research effort for this project are indicated in Table 1.



(L= 30mm, W=18mm, e= 1mm, b= 2mm)

Figure 3 Simple shear device (left), Simple shear test sample (right).

Table 1 Mechanical tests carried out in the present project

Type of test		Position of specimens in sheet	Angle (°) Direction of loading/DL	Ti-α, Plate #1	Ti-α, Plate #2
Loading path 1	Loading path 2				
Monotonic simple shear	-	#2 (∈ zone 3)	0°	☑	☑
			45°	☑	☑
			90°	☑	☑
Bauschinger Shear 40%-60%	-	#6 (∈ zone 5)	0°	☑	☑
			45°	☑	☑
			90°	☑	☑
Cyclic shear 20%-20% (3.5 cycles)	-	#5 (∈ zone 5)	0°	☑	☑
			45°	☑	☑
			90°	☑	☑

<u>X-ray diffraction</u>:

The method of determination of the pole figures by X-ray diffraction is indicated in the following: A macroscopic reference direct frame (RD, TD, ND) is associated to each sample. These three directions represent respectively the rolling, transverse and normal directions. In order to obtain the precise value of the Bragg angles relative to the planes {HKL} based on which we will determine the pole figures, a graph θ/2θ is determined step by step (step of 0.2°). For each sample, measurements of five incomplete pole figures (angle of variation limited to 80° because of problems of defocusing) allow to determine the families of planes {1 0 0}, {0 0 2}, {1 01}, {1 0 2} and {1 1 0} (five experimental pole figures are required for the calculation of the Orientations Distribution Function O.D.F). The procedure used for the measurements is as follows: θ and 2θ are fixed at the Bragg angle of the selected line of

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diffraction. The figure division of poles is of 2.5°x5° (azimuth variation) with integration of the signal received during azimutal rotation. The experimental pole figures obtained are then corrected from background noise, defocused then, standardized. The F.D.O. (complete pole figures) are then given using 2 analysis softwares (Popla ® and Labotex®). The characteristics of the diffractometer used are indicated hereafter:

-Conventional Source Co (specific hearth), goniometer 4 circles of INEL mark;

- A plane graphite monochromator (elimination of the Kß radiation), the value wavelength is: λ = 1.7902 Å (2/3 Kα1, 1/3 K α2);

- Translation of the sample on itself in order to increase the statistics of counting.

-Specific detector provided with cross slits requiring acquisition with every pole figure (better precision).

Figure 4 X-ray diffraction apparatus.

Orientation Imaging Microscopy: OIM

The technique of diffraction of the retro-diffused electrons (Electron Back Scattered Diffraction, EBSD) associated with electronic scan microscopy allows to determine the local crystallographic orientations of the grains of mono or polycrystalline materials. It is implemented by equipping a Scanning Electron Microscope (SEM) with a specific EBSD detector (Figure 5). When the electron beam is focused on the area of the sample to be analyzed, part of the electrons is retro-diffused and diffracts on the crystalline levels until providing, on a phosphorus screen, a diagram of diffraction made up of pseudo-bands known as Kikuchi bands, characteristic of the orientation of the analyzed grain. The figures formed by these bands are called diagram of diffraction of Kikuchi (Figure 6).
The electrons diffract according to two hyperboles, but under the hypothesis of small angles, one can approximate these hyperbolas by two quasi-straight and parallel lines. Each pair of lines corresponds to a family of planes of the impacted crystal and the inter-reticular distance is connected to their spacing. From this diagram of Kikuchi, one can obtain the local crystallographic orientation and the parameter of the crystal. From a practical point of view, the sample to be analyzed is tilted of 70° compared to the incidental beam, in order to collect the maximum of retro-diffused electrons on the level of the detector. Cartography of the orientations of the grains is obtained by sweeping with the electron beam the surface of the sample. This technique has a space resolution of a hundred nanometre and a precision of a few degrees. A good measure is possible on a polished sample and which has not hardened.

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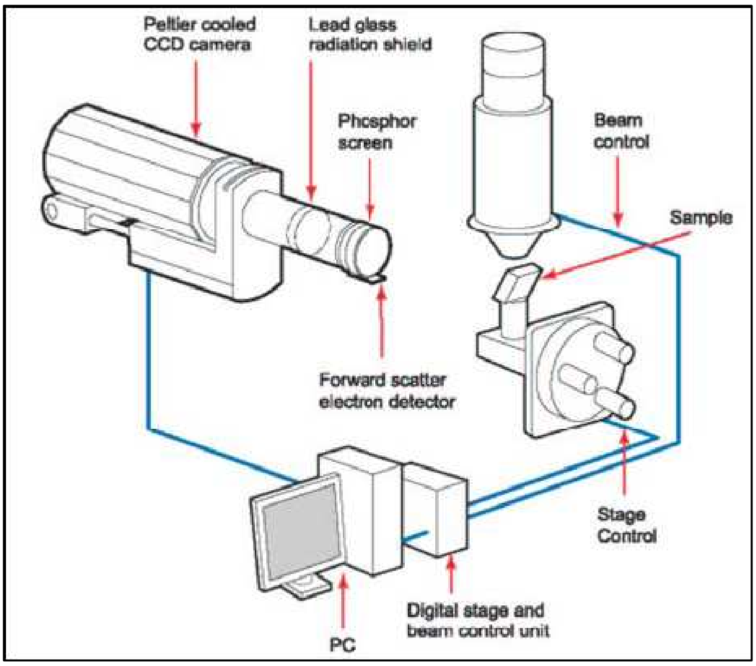

Figure 5 Schematic diagram of the EBSD installation.

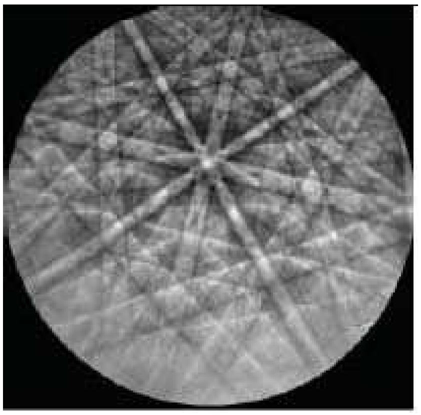

Figure 6 Kikuchi Pattern**.**

Preparation of surface for optical microscopy and SEM

The technique of preparation of the surface of the specimen must allow a good description of the grain boundaries (determination of intermediate size of grains by optical microscopy) while avoiding an artificial activation of twinning during polishing (e.g. Figure 7 in the case of pure titanium).

Before deformation all the samples were examined under optical microscopy. to characterize their initial state. Post-test (deformed) samples were analyzed for both optical microscopy and orientation imaging microscopy (OIM). The samples were first mechanically polished and then electropolished in a Struers solution. The chemical etching used on the samples was a solution of 2-6 ml HNO_3, 100 ml H_2O, and 1-3 ml HF.

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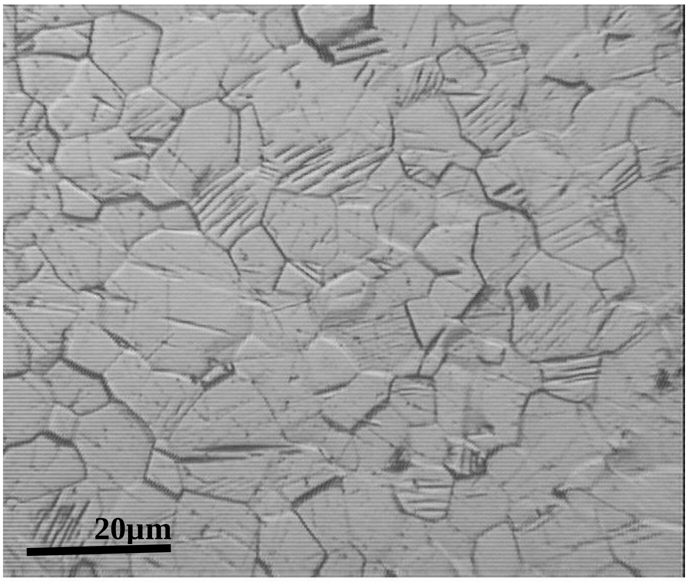

Figure 7 Micrography from optical microscope (bad preparation).

Results and Discussion

A thoroughly detailed multi-scale characterization (*i.e*. SEM observations, EBSD analysis and macroscopic texture measurements) in parallel with various mechanical tests, were carried out in order to identify the physical mechanisms at the microscopic scale (grain size and crystallographic texture evolution, intra-granular misorientation, volume twin fraction…) and determine their effects on the macroscopic behaviour e.g. Bauschinger effect, work-hardening evolution, anisotropy of the yield stress. Twinning was observed in addition to slip. The sensitivity of the volume fraction of twins to the type of loading was analyzed and its effect on the observed macroscopic hardening was quantified.

In order to investigate the through thickness properties of the material, crystallographic texture measurement by X-rays and EBSD analyses at each 1.5mm depth were carried out. It was observed that the material exhibits a strong through thickness initial anisotropy in terms of the average grain size distribution (Figure 8) and the crystallographic texture. Significant variation from the orthotropic hypothesis was observed (orientation of the c-axis with respect to the normal direction).

The mechanical behaviour was investigated using quasi-static monotonic and cyclic simple shear tests. The latter were conducted at room temperature and large strain along different directions with respect to the rolling direction (typically 0°, 45° and 90°) on the pure titanium samples. Significant stress anisotropy was observed on the stress-strain curves in terms of level of stresses and work-hardening evolution. The material displays large Bauschinger effect under reverse simple shear testing. Moreover, when the amount of plastic strain increases under cyclic loading, an abrupt work-hardening evolution is observed for a specific loading direction (45° with respect to the rolling direction).

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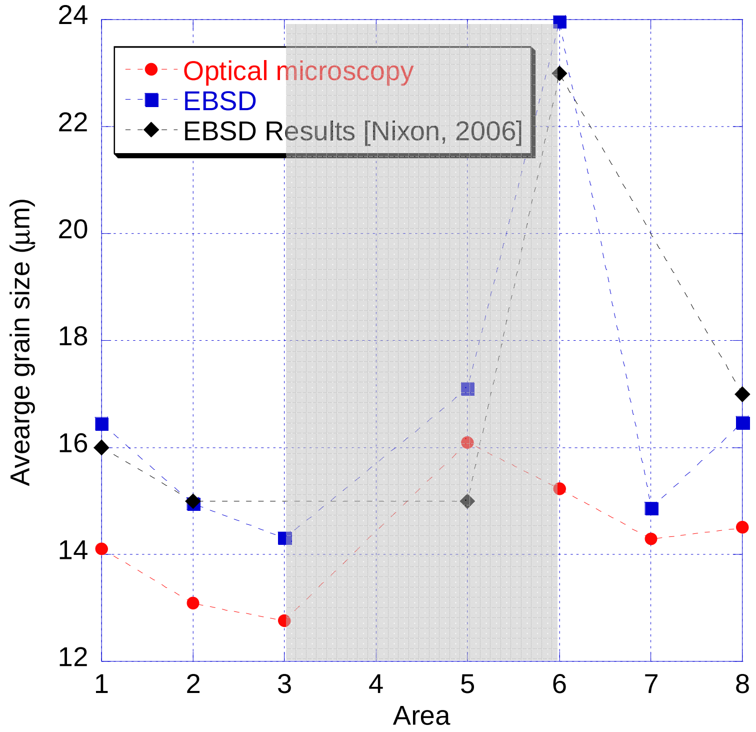

Figure 8 Variation of the mean grain size along the thickness of the plate for the as-received Plate 1 material In gray is represented the zone from where the specimens used in the mechanical tests were cut.

The stress-strain curves during monotonic loading indicate that the material displays a significant anisotropy in stress. Microstructural investigation using SEM observations on samples deformed under monotonic loadings indicate that the grain fragmentation (*i.e.* reduction in the average grain size) is found similar for the different loading directions (Figure 10). However, the hardest direction corresponds to the one where the highest twin volume fraction was observed. Consequently, twinning seems to be the dominant mechanism responsible for yielding anisotropy.

Bauschinger tests are performed for an accumulated equivalent plastic strain of $\varepsilon^p \approx 80\%$. The samples deformed by Bauschinger simple shear test along 45°/RD and 90°/RD, exhibit a significant reduction of the grain size. Conversely, along the rolling direction, the rate of grain fragmentation remains very low. However, at the macroscopic scale, the rolling and transverse directions indicate almost similar stress-strain behaviour. Analysis of the stress-strain curves shows that the 45°/RD is the hardest direction and corresponds to the highest measured twin volume fraction. Consequently, the hardening seems to be controlled in greater measure by twining than by "*Hall-Petch*" (grain fragmentation) mechanism. It is worth nothing that specimens, for the Bauschinger loading, display a different initial crystallographic texture (more orthotropic) compared to the ones used for monotonic loading. It is worth noting that for the material investigated, the Bauschinger effect is significant.

The cyclic tests up to an accumulated equivalent plastic deformation $\varepsilon^p \approx 150\%$ indicate that the direction 45°/RD is the hardest direction. Moreover, the directions 0°/RD and 90°/RD, show a very slow evolution of work-hardening which tends to reach a saturation state after few cycles. Conversely, the work-hardening seems to continue in the case of loading along

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45°/RD direction. Such macroscopic behaviour can be explained based on the microscopic observations which show that the highest volume twin fraction as well as the highest level of grain refinement are observed for the 45°/RD direction.

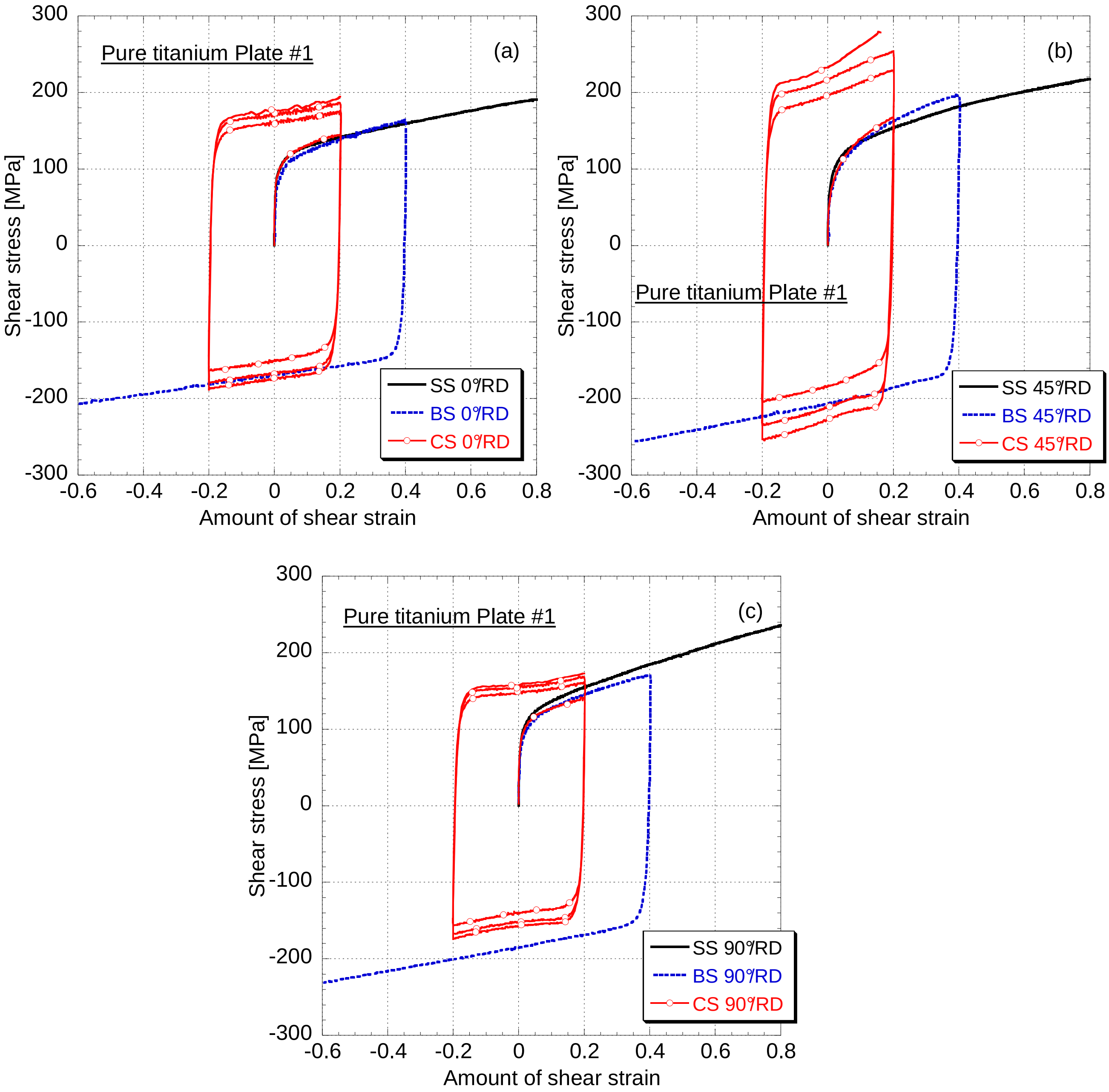

Figure 9 Mechanical behaviour under monotonic Simple Shear test (SS), Bauschinger Simple test (BS) and Cyclic Simple shear test (CS) on pure titanium Plate #1. (a) Simple shear along the rolling direction 0°/RD, (b) Simple shear along 45°/RD, (c) Simple shear along 90°/RD.

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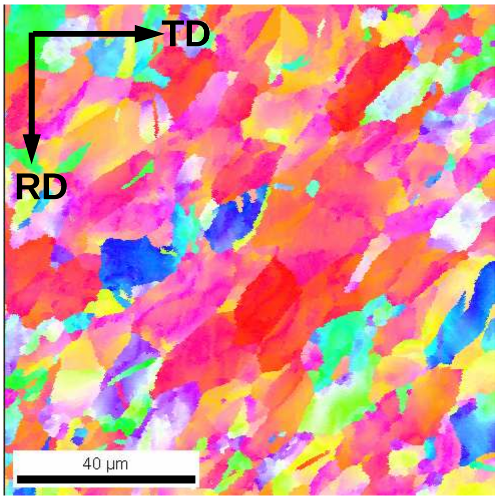
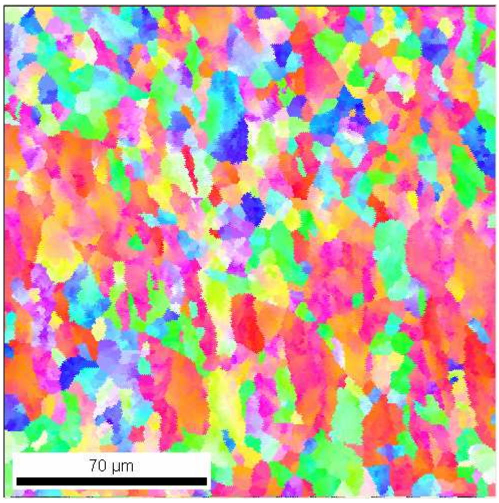
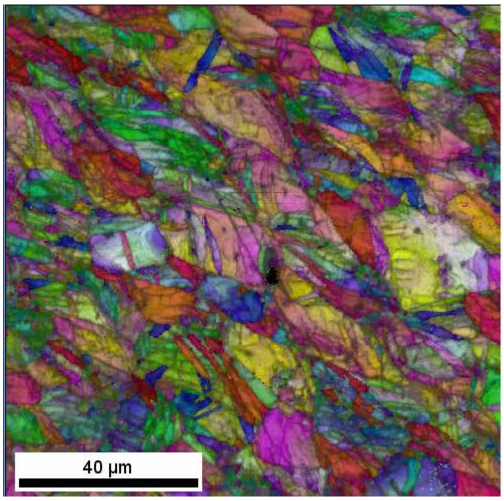


Ti-α, #2, 0°/DL



Ti-α, #2, 45°/DL



Ti-α, #2, 90°/DL

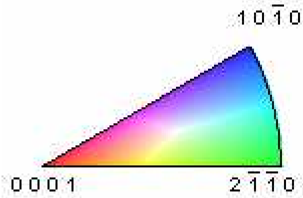


Figure 10 Orientation map of the normal direction to the sheet plane (ND) after simple shear γ=90% ($\varepsilon^p\approx$50%).

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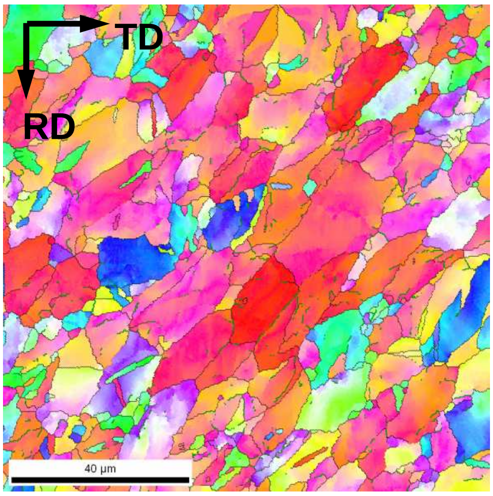

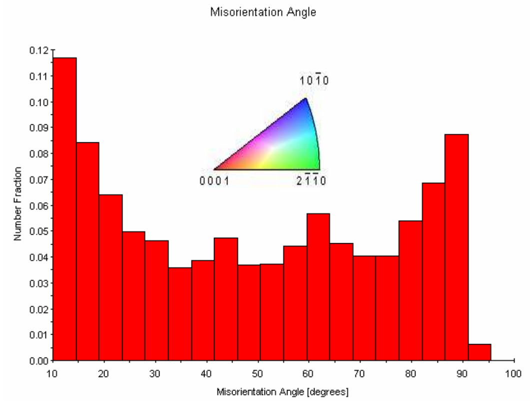

Boundaries: Rotation Angle

	Min	Max	Fraction	Number	Length
black	15°	100°	0.244	28913	5.01 mm
green	10°	15°	0.036	4225	731.79 microns

Figure 11 Orientation map of the normal direction to the sheet plane (ND) and grain misorientations distribution for pure titanium deformed by simple shear along the RD up to γ=90% ($\varepsilon^p \approx 50\%$).

Boundaries: Axis Angle

	Plane Normal	Direction	Angle	Tolerance	Phase	Match Plane	Fraction
black	-1 2 -1 0	-1 2 -1 0	94.8°	1°	Titanium (Alpha)	1 0 -1 2	0.001
orange	-1 1 0 0	-1 1 0 0	34.8°	10°	Titanium (Alpha)	1 1 -2 1	0.005
black	-1 1 0 0	-1 1 0 0	64.3°	8°	Titanium (Alpha)	1 1 -2 2	0.011
blue	-1 2 -1 0	-1 2 -1 0	57°	8°	Titanium (Alpha)	1 0 -1 1	0.002
black	3 0 -3 -5	1 0 -1 -1	85°	8°	Titanium (Alpha)	1 0 -1 2	0.003
black	2 2 -4 -5	2 2 -4 -3	85°	8°	Titanium (Alpha)	1 0 -1 2	0.012
black	0 -19 19 16	0 -2 2 1	85°	8°	Titanium (Alpha)	1 0 -1 2	0.003
black	1 -2 1 0	1 -2 1 0	85°	8°	Titanium (Alpha)	1 0 -1 2	0.018

Tableau 1 Type of twin family and volume fraction of each twin family observed in pure titanium deformed by simple shear along the RD up to γ=90% ($\varepsilon^p \approx 50\%$).

Conclusions

The present work focused on the multi-scale investigation of the mechanical behaviour of pure titanium Ti-α. The material was deformed by monotonic and cyclic simple shear test up to large strains. The mechanical tests were performed along three directions: rolling, transverse and along 45° from the rolling direction to quantify its anisotropy and the anisotropy evolution. Stress-strain curves indicate that the material displays significant in plane anisotropy in term of stress level. SEM investigations reveal that:

- For monotonic simple shear conditions, the hardest direction corresponds to the one where a significant amount of twins was observed. The mean grain size remains constant (in comparison with the as-received material).
- The occurrence of twinning seems to be related to an increase in work-hardening rate.

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- For Bauschinger simple shear loading, the hardest direction corresponds to the 45° with respect to the rolling direction. Loading in this direction as well as in the transverse direction, induces significant grain fragmentation (about 50% grain size reduction). Such a significant grain fragmentation may be related to twin activation. Indeed, EBSD analyses reveal that significant twins are present in the sample deformed along 45°/RD.
- After cyclic loading along 45°/RD, the level of flow stresses increases with the number of cycles. As for the Bauschinger loading, a significant grain size reduction is observed. However, the measured volume fraction of twins is lower when compared to the value obtained for the Bauschinger simple shear test, which indicates that some untwining may have occurred during cyclic loading.

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